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Adsorption and inhibition action of a novel green inhibitor on aluminium and 6063 aluminium alloy in 1.0 M H_3PO_4 solution

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Abstract

The corrosion inhibition characteristics of *Garcinia indica* extract (GIE) on the corrosion of aluminium and 6063 aluminium alloy in 1.0M phosphoric acid solution were studied by using potentiodynamic polarization using Tafel extrapolation technique and electrochemical impedance spectroscopy (EIS) techniques at different temperatures and at different concentrations of inhibitor. Kinetic parameters and thermodynamic parameters were evaluated and discussed in detail. Inhibition efficiency increased with extract concentration and increased with temperature. The adsorption of GIE on metal surface obeyed Langmuir adsorption isotherm. GIE acted as mixed inhibitor in phosphoric acid solution and followed chemisorption on the surface of both material. Results obtained from Tafel extrapolation technique and by A.C electrochemical impedance spectroscopy (EIS) technique were in good agreement with one another. This study highlights the utility of GIE extract as a potential green inhibitor for corrosion control of Al and 6063 Al alloy in 1.0M phosphoric acid environment.

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Keywords: Aluminium, 6063 Aluminium alloy; Garcinia indica; Tafel extrapolation technique; EIS technique

1. Introduction

Corrosion studies of Al and Al alloys have received considerable attention by researchers because of their industrial applications and economic considerations. The known hazardous effects of most synthetic organic inhibitors and the need to develop cheap, non-toxic and environmentally benign process now urged researchers to focus on the use of environmentally friendly and ecologically acceptable, plant products as corrosion inhibitor by Deepa et al. (2013). Till date lots of plant products have been used as effective corrosion inhibitor for steel and Al observed in a review article by Raja et al. (2008). As a contribution to the current interest of environment friendly

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corrosion inhibitors, our present work investigates the inhibiting effect of *Garcinia indica* on corrosion behavior of Al and 6063 Al in alloy in 1.0 M H_3PO_4medium.

2. Experimental

2.1. Material

The Al and 6063 Al alloy specimen were used in the study. The composition of the Al is 99.6% pure and 6063 Al alloy is Si (0.412%), Fe (0.118%), Cu (0.0570%), Mg (0.492%), Al (Balance).

2.2. Medium and Inhibitor

Phosphoric acid of exactly 1.0M was prepared by diluting the standard solution of 2.0M phosphoric acid with double distilled water. Seeds of *Garcinia indica* were finely powdered and aqueous extract was prepared according to Handa et al. (2008). The aqueous extract was filtered and the filtrate was evaporated to dryness to get solid residue. FTIR spectrum of the solid residue was taken using KBr pellet technique. The aqueous solution of the inhibitors of strength was prepared and used for the experiment. The experiments were carried out at temperatures 30℃, 35℃, 40℃, 45℃ and 50℃, (±0.5℃) in a calibrated thermostat.

2.3. Electrochemical measurements

Electrochemical measurements were carried out using an electrochemical work station (CH600D-series, U.S. Model with CH instrument beta software). The electrochemical cell used was a conventional three-electrode compartment having glass cell with a platinum counter electrode, a saturated calomel electrode (SCE) as reference and working electrode was made of Al and 6063 Al alloy.

3. Results and discussion

3.1 Fourier transform infrared (FTIR) spectroscopy

Fig.1 shows the FTIR spectroscopy of GIE. –OH stretching frequency appears at 3396.41 cm^{-1}. The aromatic stretching frequency appears at 2923.88 cm^{-1}. –C=O– stretching frequency is at 1785.96 cm^{-1}. –C=C– stretching frequency at 1608.52 cm^{-1}. $-CH_2$ bending frequency 1446.51 cm^{-1}. –C–H bending frequency at 1282.57 cm^{-1}. –C=C– bending frequency at 1099.35 cm^{-1}.

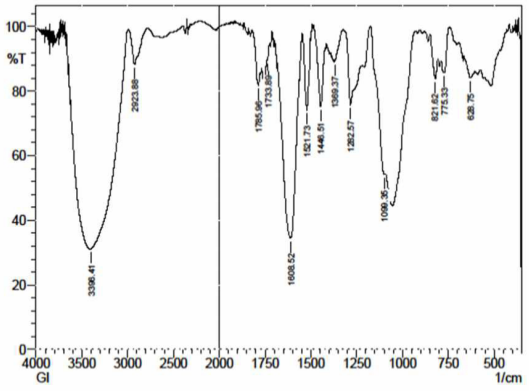


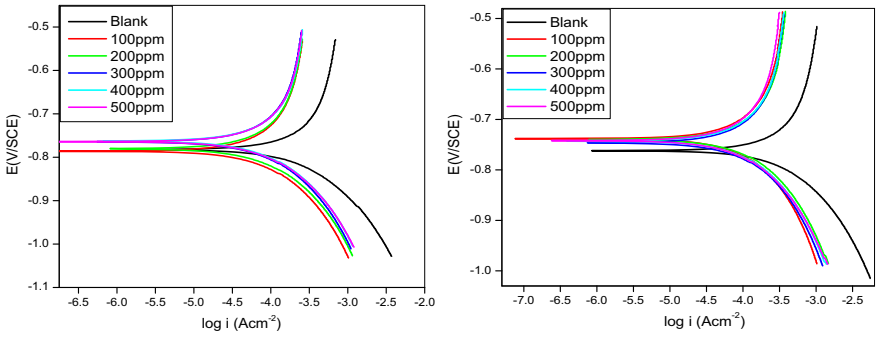
Fig. 1. FTIR spectra of solid residue of GIE.

3.2 Tafel polarization

The Tafel plots for the corrosion of Al and 6063 Al alloy specimen in 1.0 M phosphoric acid in the presence of different concentrations of GIE, at 30℃ are shown in the Fig.2a and Fig.2b respectively. Similar plots were obtained at different temperatures studied. The potentiodynamic parameters such as corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (b_c and b_a), corrosion current density (i_{corr}) and the inhibition efficiency values η (%) were obtained from Tafel plots. The results of Tafel plots are given in Tables 1. The maximum quantity of the inhibitor reported corresponds to the optimum concentration of the inhibitor.

Table 1. Electrochemical parameters obtained from Tafel polarization measurements of Al and 6063 Al alloy in 1.0 M phosphoric acid at various concentrations of GIE.

T (℃)	Aluminium							6063 Aluminium Alloy					
	Conc. of inhibitor (ppm)	E_{corr} (mV vs SCE)	i_{corr} (m A cm^{-2})	β_a (m V dec^{1})	$-\beta_c$ (m V dec^{1})	υ_{corr} (mm y^{-1})	η (%)	E_{corr} (mV vs SCE)	i_{corr} (m A cm^{-2})	β_a (m V dec^{-1})	$-\beta_c$ (mV dec^{-1})	υ_{corr} (mm y^{-1})	η (%)
30	Blank	-783	0.26	336	674	2.82	-----	-762	0.39	334	672	4.21	-----
	100	-764	0.15	358	637	1.58	43.93	-738	0.19	370	599	2.10	50.02
	200	-780	0.13	356	641	1.42	49.65	-740	0.16	368	607	1.76	58.18
	300	-764	0.12	358	637	1.35	52.06	-746	0.15	370	598	1.63	61.32
	400	-763	0.10	359	642	1.11	60.78	-743	0.12	371	610	1.26	70.1
	500	-764	0.09	360	653	1.00	64.48	-742	0.10	360	607	1.09	74.12
35	Blank	-790	0.30	348	680	3.28	-----	-778	0.51	343	658	5.50	-----
	100	-766	0.16	365	646	1.70	48.34	-737	0.21	358	602	2.34	57.53
	200	-763	0.14	362	641	1.51	53.90	-743	0.19	379	601	2.08	62.18
	300	-766	0.13	365	646	1.39	57.54	-741	0.17	393	590	1.83	66.84
	400	-758	0.11	360	637	1.16	64.68	-858	0.13	419	592	1.43	74.02
	500	-771	0.10	361	647	1.04	68.23	-736	0.11	365	603	1.21	77.93
40	Blank	-794	0.41	353	658	4.48	------	-788	0.69	350	643	7.54	------
	100	-765	0.19	378	608	2.06	53.96	-736	0.28	377	575	3.01	60.08
	200	-765	0.17	375	609	1.81	59.54	-740	0.22	386	576	2.40	68.15
	300	-765	0.16	378	601	1.70	62.08	-741	0.20	381	578	2.13	71.73
	400	-759	0.12	371	607	1.34	70.12	-743	0.14	375	577	1.53	79.74
	500	-764	0.11	375	612	1.17	73.97	-744	0.12	361	573	1.32	82.56
45	Blank	-796	0.55	358	620	6.04	-----	-805	0.96	369	597	10.46	------
	100	-765	0.23	377	582	2.53	58.07	-741	0.33	405	558	3.65	65.14
	200	-749	0.20	376	581	2.14	64.56	-748	0.27	394	555	2.89	72.37
	300	-765	0.17	377	582	1.84	69.47	-740	0.20	409	551	2.21	78.85
	400	-761	0.15	369	578	1.65	72.73	-747	0.18	398	556	1.98	81.08
	500	-759	0.12	375	579	1.30	78.54	-748	0.11	386	551	1.20	88.54
50	Blank	-805	0.76	363	589	8.26	-----	-809	1.35	379	560	14.76	-----
	100	-765	0.27	380	551	2.90	64.95	-749	0.37	433	531	4.00	72.89
	200	-763	0.24	382	557	2.59	68.59	-757	0.32	428	536	3.51	76.23
	300	-765	0.20	380	551	2.23	73.04	-753	0.24	432	529	2.57	82.56
	400	-761	0.16	385	552	1.79	78.35	-761	0.16	418	534	1.76	88.09
	500	-764	0.13	388	550	1.40	83.06	-759	0.09	417	536	1.03	93.04



(a) (b)

Fig. 2. Tafel polarization curves for the corrosion of (a) Al and (b) 6063 Al alloy in 1.0 M phosphoric acid containing different concentrations of GIE at 30°C.

From Fig. 2 and from Table 1; it can be observed that the i_{corr} decreased significantly after the addition of GIE. The η% increased with the increase in the inhibitor concentration, up to an optimum value. The value of cathodic Tafel slope (b_c) and anodic Tafel slope (b_a) does not change significantly with the increase in GIE concentration. This indicates that the addition of GIE does not change the hydrogen evolution reaction mechanismand its non-interference of inhibitor in the mechanism of anodic reaction observed by Abiola et al. (2008). From Tables 1, it is obvious that there is no appreciable shift in the corrosion potential value (E_{corr}) after the addition of GIE, which indicates that GIE is a mixed type inhibitor observed by Li et al. (2008).

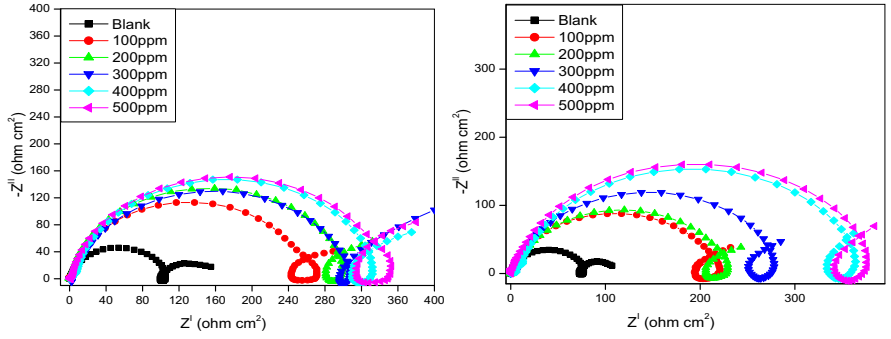
3.3 Electrochemical impedance spectroscopy (EIS) studies

The impedance plots recorded for the corrosion of Al and 6063 Al alloy in the presence and absence of inhibitor were modeled by using the equivalent circuit, which consists R_s is the solution resistance and R_{ct} is the charge transfer resistance. R_L and L represent the inductive elements. This also consists of constant phase element; CPE (Q) in parallel to the series capacitors C_1, C_2 and series resistors R_1, R_2, R_L and R_{ct}. R_L is parallel with the inductor L. The polarization resistance R_p and double layer capacitance C_{dl} can be calculated from equations (1) and (2)

$$R_p = R_L + R_{ct} + R_1 + R_2 \quad (1)$$

$$C_{dl} = C_1 + C_2 \quad (2)$$

Fig. 3a and Fig. 3b shows the Nyquist plots for the corrosion of Al and 6063 Al alloy in 1.0 M phosphoric acid solution at 30°C in the presence of different concentrations of GIE. Similar plots were obtained at all other temperatures. The results of EIS studies for the corrosion inhibition of Al and 6063 Al alloy in presence of GIE are summarized in Table 2.



(a) (b)

Fig. 3. Nyquist plots of Al and 6063 Al alloy in 1.0 M phosphoric acid at 30°C in the presence of different concentrations of GIE.

As can be seen from Fig. 3 the Nyquist plots are semi-circular in the presence as well as in the absence of inhibitor. This indicates that the corrosion of Al and 6063 Al alloy is controlled by a charge transfer process in the presence and absence of the inhibitor. Similar impedance plots have been reported in literature for the corrosion of Alobserved by Trowsdale et al.(1996). According to Brett et al. (1990) (1992), the capacitive loop is corresponding to the interfacial reactions, particularly, the reaction of Al oxidation at the metal/oxide/electrolyte interface. The other explanation offered to the high frequency capacitive loop is the oxide film itself. The origin of the inductive loop has often been attributed to surface or bulk relaxation of species in the oxide layer observed by Wit et.al. (1996). The inductive loop may be related to the relaxation process obtained by adsorption and incorporation of phosphate ions on and into the oxide film. In order to fit and analyze the EIS data, the experimental impedance results are simulated to pure electronic models that can verify or role out mechanistic models and enable the calculation of numerical values corresponding to the physical and/or chemical properties of the electrochemical system under investigation. However, the impedance plots recorded for the corrosion of the Al and 6063 Al alloy in the presence of GIE were modeled by using the equivalent circuit depicted in Fig.4. The circuit fitment was done by ZSimpWin software version 3.21. The results recorded from Table 2 indicated the increase of R_p value with the increase in the concentrations of the inhibitor. This suggests that the amount of the inhibitor molecules adsorbed on the electrode surface increases as the concentration of GIE increases. The decrease in C_{dl} could be attributed to the decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, signifying that the GIE molecules act by adsorption at the interface of metal/solution.

Table 2. Electrochemical parameters obtained from EIS measurements of Al and 6063 Al alloy in 1.0 M phosphoric acid at various concentrations of GIE.

T (℃)	Aluminium				6063 aluminium alloy		
	Conc. of inhibitor (ppm)	R_p (Ω cm^2)	CPE (μ F cm^{-2})	η (%)	R_p (Ω cm^2)	CPE (μ F cm^{-2})	η (%)
30	Blank	156.30	38	------	101.70	69	------
	100	268.76	30	41.84	198.48	64	48.76
	200	300.43	29	47.97	238.19	60	57.30
	300	316.03	28	50.54	257.93	58	60.57
	400	388.52	26	59.77	335.13	55	69.65
	500	430.03	25	63.65	387.97	50	73.79
35	Blank	130.20	45	------	75.90	78	------
	100	244.03	44	46.65	170.71	65	55.54
	200	274.43	40	52.56	192.69	61	60.61
	300	298.64	39	56.40	220.89	59	65.64
	400	360.63	38	63.90	284.15	57	73.29
	500	401.82	36	67.60	335.91	55	77.40
40	Blank	94.50	58	------	54.00	90	------
	100	199.26	57	52.57	128.27	88	57.90
	200	227.56	55	58.47	162.54	86	66.78
	300	243.21	53	61.14	184.02	84	70.65
	400	310.27	50	69.54	259.54	81	79.19
	500	357.04	48	73.53	302.63	77	82.16
45	Blank	70.20	66	------	36.80	117	------
	100	162.42	64	56.78	101.57	115	63.77
	200	193.08	63	63.64	129.19	99	71.51
	300	224.94	60	68.79	170.00	91	78.35
	400	252.43	57	72.19	190.50	83	80.68
	500	322.12	56	78.21	317.12	78	88.40
50	Blank	50.20	75	------	24.20	129	------
	100	139.22	73	63.94	83.27	125	70.94
	200	155.82	70	67.78	95.81	113	74.74

	300	182.20	68	72.45	132.76	101	81.77
	400	227.87	67	77.97	197.19	92	87.73
	500	292.34	65	82.83	341.70	85	92.92

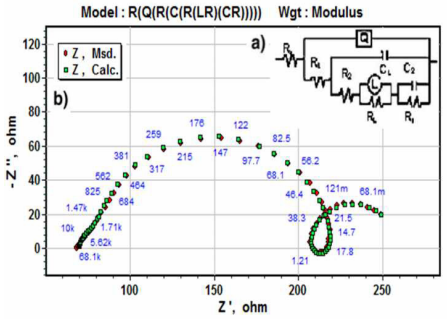
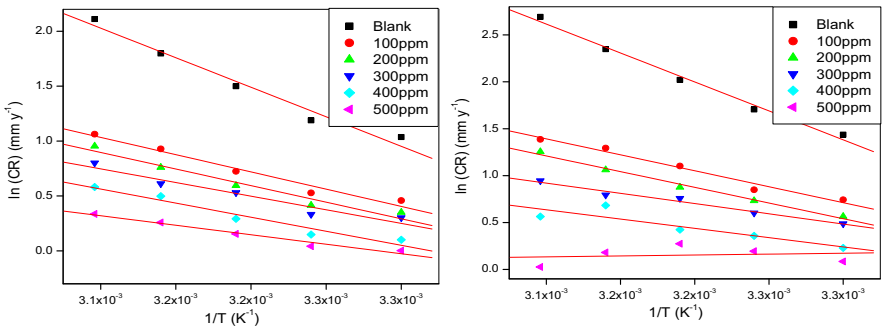


Fig. 4. Equivalent circuit used to fit the experimental EIS data for the corrosion of Al and 6063 Al alloy specimen in the presence of different concentrations of GIE.

3.4 Effect of temperature

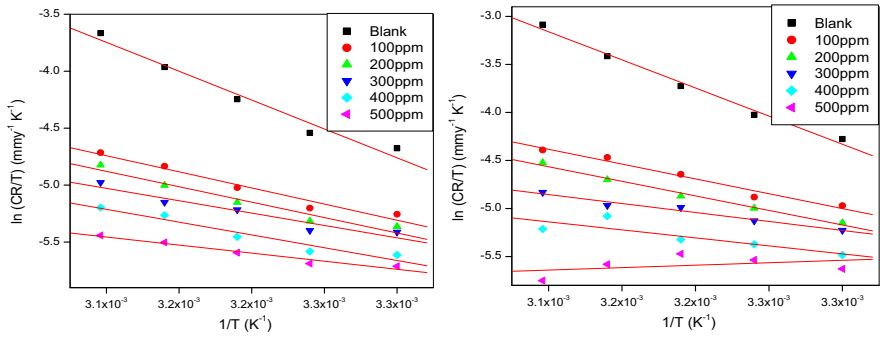
The study on the effect of temperature on the corrosion rate and inhibition efficiency facilitates the calculation of kinetic and thermodynamic parameters for the inhibition and the adsorption processes. The increase in inhibition efficiency with the increase in temperature may be attributed to the lower dissolution rates of Al and 6063 Al alloy at elevated temperature. The increase in inhibition efficiency with the increase in temperature is also suggestive of chemisorption of the inhibitor molecules on the metal surface observed by Abdel-Gaber, et al. (2006). The Arrhenius plots for the corrosion of Al and 6063 Al alloy in the presence of different concentrations of GIE in 1.0 M phosphoric acid are shown in Fig. 5a and Fig. 5b.



(a) (b)

Fig. 5. Arrhenius plots for the corrosion of Al and 6063 Al alloy in 1.0 M phosphoric acid containing different concentrations of GIE.

The plots of $\ln(\upsilon_{corr}/T)$ versus 1/T for the corrosion of Al and 6063 Al alloy in the presence of different concentrations of GIE in 1.0 M phosphoric acid are shown in Fig. 6a and Fig. 6b.



(a) (b)

Fig. 6. Plots of ln (υ_{corr}/T) vs 1/T for the corrosion of Al and 6063 Al alloy in 1.0 M phosphoric acid containing different concentrations of GIE.

The calculated values of activation parameters are recorded in Table 3. The decrease in apparent activation energy may be interpreted as due to chemical adsorption of the inhibitor. Large negative values of entropies show that the activated complex in the rate determining step is an association rather than dissociation step meaning that a decrease in disordering takes place on going from reactants to the activated complex observed by Oguzie et al. (2007).

Table 3. Activation parameters for the corrosion of Al and 6063 Al alloy in 1.0 M phosphoric acid containing different concentrations of GIE.

Activation parameters	Aluminium						6063 aluminum alloy					
	Inhibitor concentration (ppm)						Inhibitor concentration (ppm)					
	0	100	200	300	400	500	0	100	200	300	400	500
E_a(kJ mol^{-1})	44.74	26.1	25.1	21.2	20.6	14.3	50.2	28.2	27.8	18.1	16.4	1.62
ΔH_a(kJ mol^{-1})	42.13	23.5	22.5	18.6	18.0	11.7	47.6	25.6	25.2	15.5	13.8	4.2
ΔS_a(J mol^{-1} K^{-1})	-98.0	-164.0	-168.3	-183.0	-183.4	-206.4	-72.9	-154.4	-167.3	-189.8	-197.3	-257.5

3.5 Adsorption behavior

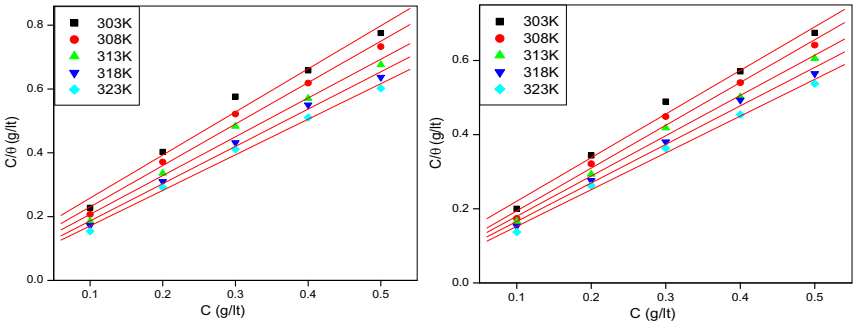
The information on the interaction between the inhibitor molecules and the metal surface can be provided by adsorption isotherm. The values of θ at different concentrations of inhibitor in the solution (C_{inh}) were applied to various isotherms including Langmuir, Temkin, Frumkin and Freundlich isotherms. It was found that the data fitted best with the Langmuir adsorption isotherm, which is given by the relation:

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K} \quad (3)$$

where K is the adsorption/desorption equilibrium constant, C_{inh} is the corrosion inhibitor concentration in the solution, and θ is the surface coverage. The plot of C_{inh}/θ versus C_{inh} gives a straight line with an intercept of 1/K. The Langmuir adsorption isotherms for the adsorption of GIE on the Al and 6063 Al alloy surface are shown in Fig.7a and Fig. 7b. The plots are linear, with an average correlation coefficient (R^2) of 0.99. The linear regression coefficients are close to unity suggesting that the adsorption of GIE obeys Langmuir's adsorption isotherm and there is negligible interaction between the adsorbed molecules observed by Trowsdale et al. (1996). Fig. 8a and Fig. 8brepresents the plot of free energy versus temperature for Al and 6063 Al alloy in phosphoric acid. Thermodynamic parameters for the adsorption of GIE on Al and 6063 Al alloy in phosphoric acid at different temperatures are listed in Table 4.

Table 4. Thermodynamic parameters for the adsorption of GIE on Al and 6063 Al alloy in 1.0 M phosphoric acid at different temperatures.

T (K)	Aluminium			6063 aluminum alloy		
	$-\Delta G^{\circ}_{ads}$ (kJ mol^{-1})	ΔH°_{ads} (kJ mol^{-1})	ΔS°_{ads} (J mol^{-1} K^{-1})	$-\Delta G^{\circ}_{ads}$ (kJ mol^{-1})	ΔH°_{ads} (kJ mol^{-1})	ΔS°_{ads} (J mol^{-1} K^{-1})
303	21.20			21.65		
308	22.07			22.68		
313	22.85	29.71	-148.90	23.32	25.34	-136.29
318	23.76			24.06		
323	24.56			24.85		



(a) (b)

Fig. 7. Langmuir adsorption isotherms for the adsorption of GIE on Al and 6063 Al alloy in 1.0 M phosphoric acid at different temperatures.

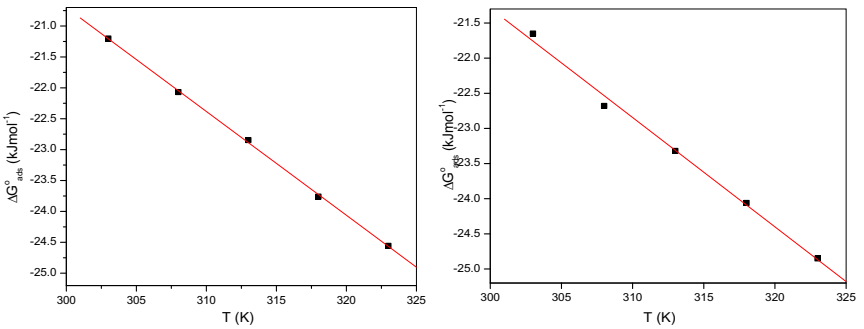
The values of standard free energy (ΔG^{0}_{ads}) of adsorption are related to K by the relation shown in equation.

$$K = \frac{1}{55.5} \exp\left(\frac{-\Delta G^{\circ}_{ads}}{RT}\right) \quad (4)$$

where the value 55.5 is the concentration of water in solution in mol dm^{-3}, R is the universal gas constant, and T is absolute temperature. The standard enthalpy of adsorption (ΔH^{0}_{ads}) and standard entropy of adsorption (ΔS^{0}_{ads}) were calculated from the plots of (ΔG^{0}_{ads}) vs T as per the equation (5), which is obtained from the plots of (ΔG^{0}_{ads}) vs T.

$$\Delta G^{\circ}_{ads} = \Delta H^{\circ}_{ads} - T\Delta S^{\circ}_{ads} \quad (5)$$

The negative values of ΔG^{0}_{ads} indicate the spontaneity of the adsorption process and the stability of the adsorbed layer on the metal surface observed by Tao et al. (2011). Generally, value of ΔG^{0}_{ads} up to -20 kJ mol^{-1} is consistent with the electrostatic interaction between the charged molecules and the charged metal (physical adsorption) while the value more negative than -40 kJ mol^{-1} involve sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a co-ordinate type of bond (chemisorption) observed by Shivakumar et al. (2013). In the present study, the values of ΔG^{0}_{ads} are above -20 kJ mol^{-1}. Even though it is suggestive of physical adsorption, we need to consider the value of ΔH^{0}_{ads} for the endothermic reaction, as in case of the present investigation. ΔG^{0}_{ads} and inhibition efficiency increase with the increase in temperature, indicates that the adsorption of GIE on the Al surface in phosphoric acid are favored at high temperature and hence can be considered to be predominantly chemisorption. The positive value of enthalpy of adsorption indicates that the adsorption of the inhibitor molecule is an endothermic process, which suggests chemisorption. The ΔS^{0}_{ads} value is large and negative; indicating that decrease in disordering takes place on going from the reactant to the adsorbed species. This can be attributed to the fact that adsorption is always accompanied by decrease in entropy observed by Shivakumar et al. (2013).

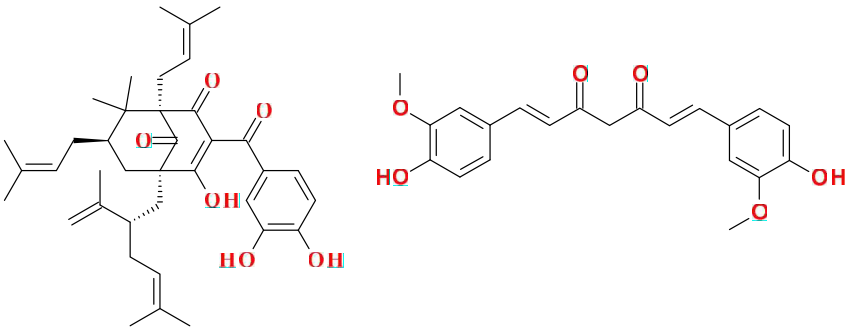


(a) (b)

Fig 8. Plots of (ΔG^0_{ads}) vs T for Al and 6063 Al alloy in 1.0 M phosphoric acid in presence of GIE.

3.6 Explanation for inhibition

Al and Al alloy in acidic solutions undergo corrosion by the dissolution of the Al ions from the oxide free metal surface into the solution and the principal cathodic process is thedischarge of hydrogen ions to produce hydrogen gas. The 6063 Al alloy consists of Si and Mg as major alloying elements with very small amount of Fe. According to Kaoru Mizuno et al. (2001) the alloy can thereby be considered as a four phase material: the Al matrix, Mg_2Si, AlFeSi and Si. The electrochemical properties of the four phases differ markedly. The equilibrium potentials for the Al matrix, Mg_2Si, AlFeSi and Si particles are -1080,-1230, -720 and -500 mV (SHE), respectively. Thus, the AlFeSi and Si particles are expected to be cathodic when compared to the Al matrix. The surface reaction products formed on the four phases during oxidation were characterized by Nylund et al. (1998). These phases contribute to the enhanced corrosion rate as well as corrosion inhibition of 6063 aluminium alloy in comparison with aluminium (99.6%). The inhibitor molecules of GIE have the electron rich environment in phenyl group (Fig.9) due to π-electrons of double bonds that can be adsorbed well on the sample surface. Also these groups are electron donating groups containing oxygen atom as an active site. GIE inhibits the corrosion primarily through its adsorption and subsequent formation of a barrier film on the metal surface.



Garcinol **Curcumin**

Fig. 9.Structure of active constituents of GIE.

Adsorption may take place by organic molecules at metal/solution interface due to bonding between the charged molecules and charged metal, interaction of unshared electron pairs in the molecule with the metal, interaction of π -electrons with the metal or the combination of the above. In acidic medium like the one in the present investigation, the metal surface is positively charged. This would cause the negatively charged phosphate ions to become adsorbed

on the metal surface, making the metal surface negatively charged. The negative charge centers of the GIE molecules containing a lone pair of electrons and/or π electrons can interact with the anodic sites on the metal surface and get adsorbed. Chemisorption is also possible by the donor - acceptor interactions between π electrons of the aromatic ring and metal surface, providing another mode of protection. The presence of GIE in the protonated form and the presence of negative charge centers on the molecule are also responsible for the mutual interaction of inhibitor molecules on the metal surface. This is reflected in the deviation of slopes of Langmuir adsorption isotherms.

4. Conclusions

- GIE acts as a good eco-friendly green inhibitor for the corrosion control of Al and 6063 Al alloy in phosphoric acid medium.
- Inhibition efficiency of the GIE was efficient for 6063 Al alloy when compared with Al (99.6 %).
- Inhibition efficiency of the GIE increases with increase in the concentration of the inhibitor and increase with increase in the temperature for both the materials.
- GIE acts as a mixed type of inhibitor and obeys Langmuir adsorption isotherm and the adsorption is through chemisorption.
- Results obtained by Tafel polarization and EIS techniques are in good agreement with one another.

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